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(54) Title: ENCAPSULATE OF ACTIVE MATERIAL IN ALGINATE MATRIX

(57) Abstract

A hydrated encapsulate of active material comprises an emulsion or dispersion of active material in an alginate matrix. The invention finds particular application in dental flavour encapsulates for use in toothpastes.

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ENCAPSULATE OF ACTIVE MATERIAL IN ALGINATE MATRIX

Field of the Invention

This invention concerns encapsulation and relates to encapsulates of active material, a method of making the encapsulates, and products, particularly toothpastes, incorporating the encapsulates.

Background to the Invention

Encapsulation is a well known technique for presenting active materials such as flavour, fragrance, colouring materials etc. Encapsulate systems fall into two main types: those in which the active material is surrounded by a wall or barrier; and those in which the active material is encapsulated in the matrix of a material. A common form of matrix encapsulate is based on a matrix of cross-linked alginate gels.

Alginates are naturally occurring colloids which are normally extracted from brown seaweed (Phaeophycea), which are used mainly in the form of sodium salts. Sodium alginates give very viscous solutions with water and can be irreversibly cross-linked to form thermally stable gels by interaction with divalent or trivalent metal ions, typically calcium ions. Using this interaction, active materials may be encapsulated or entrapped in an alginate gel matrix. The colloid is principally alginic acid which is made up of three kinds of polymer segments, one segment containing D-mannuronic acid units; a second segment containing L-guluronic acid; and a third segment containing alternating D-mannuronic and L-guluronic acid residues, joined by 1:4 glycosidic linkages. The proportions of the three polymer segments in alginic acid samples give rise to different properties, for example, the alginate from Laminaria hyperborea, with a large percentage of polyguluronate segments, forms rigid, brittle gels which tend to undergo syneresis.

There is a large amount of literature in the area of dental flavour encapsulation, not only to provide flavour stability but also to develop a unique flavour delivery system which provides a perceivable sensory effect and longevity of the flavour in the mouth. To this end, numerous systems have been tested but there are currently no toothpastes in the market place which utilise a flavour encapsulation system.

US 4389419 (Lim) concerns capsules of oil soluble nutrients such as vitamins which are formed by making an emulsion of sodium alginate with the nutrient and with optional alcohol-insoluble polysaccharide filler or extender such as dextran, sodium carboxy methyl cellulose, methyl cellulose, dextrins and some soluble starches and sodium carboxy cellulose, and adding the emulsion as droplets to an alcoholic solution containing calcium chloride to give solid capsules. The resulting capsules or beads are dried. The wall of the capsule comprises the matrix of water-insoluble multivalent cation-containing gel and filler, and within the matrix is a plurality of compartments containing oil droplets shielded from atmospheric exposure. Column 2 lines 48 to 50 state that advantageously no emulsifying agents need to be employed since the alginate (which is present at high levels) effectively serves this function. No uses of the capsules are specified.

EP 0202819A (Warner Lambert) discloses encapsulates formed by mixing alginate with active ingredient, particularly to produce a dispersion, and crosslinking the alginate by exposure to divalent cations, for example by spraying the dispersion into calcium chloride to produce particles eg up to 10 micron in diameter. The resulting particles are dried. The active ingredients may be flavouring, colouring agent, fragrance etc. The encapsulates may be used particularly in chewing gum, but also in confectionary, foods, pharmaceuticals, toothpaste etc. No details are given of toothpaste formulations.

EP 0747037A (Sara Lee) discloses toothpaste including microspheres 0.1-4mm in diameter of zinc-stabilised alginate. The microspheres can contain ingredients such as enzymes, abrasives, flavour, colour, bactericides etc. The microspheres are prepared by adding droplets of alginate solution to a zinc salt solution such as zinc chloride. The alginate solution preferably includes a surfactant such as polyethylene glycol (PEG) or ethoxylated glycerol, for unspecified purpose. The materials are not in the form of an emulsion.

Summary of the Invention

In one aspect the present invention provides a hydrated encapsulate of active material, comprising an emulsion or dispersion of active material in an alginate matrix.

The present invention is particularly directed to provide a flavour release system, preferably for toothpaste - one which can be incorporated into water-based toothpastes, is stable on storage yet releases during brushing in the mouth. The invention also provides

for a visual effect in the toothpaste, contributing to its appeal, which can enhance both flavour intensity and longevity. There is also the opportunity to deliver novel flavour effects e.g. change of flavour on brushing.

It has been found that by having the reagents in the form of an emulsion or dispersion, and by having the encapsulates in hydrated form, benefits in terms of product stability during storage and use can be obtained. In particular, the properties of the active material are less likely to deteriorate with time and the encapsulates are more stable in possibly hostile environments of use.

The active material may be selected from a wide range of different materials, depending on the intended use of the encapsulate, including, for example, flavouring materials, including dental flavours and food flavours, fragrance materials, colouring materials, enzymes, therapeutic agents such as anti-bacterial agents, anti-caries agents, etc. The active material is preferably substantively insoluble in water, and/or preferably sufficiently large (eg preferably having a molecular weight of at least 5000) to be retained by the alginate. Suitable materials are well known. A mixture of active materials may be used.

The active material is present in relatively large amounts, typically constituting up to 60% by weight of the total weight of the encapsulates. The active material is typically present in an amount in the range 10 to 60%, preferably 20 to 60%, eg 25 to 30% by weight of the total weight of the encapsulate. References to the total weight of the encapsulate mean the weight of all encapsulate ingredients, including water etc.

The alginate is conveniently in the form of a sodium salt, and is preferably an alginate having a high percentage of polyguluronic acid units (known as high G alginates) as these produce encapsulates with improved stability on storage. Suitable alginates are commercially available, and include, for example, the high G alginate Manugel DMB (Manugel is a Trade Mark) supplied by Nutrasweet Kelco. The aforementioned alginates provide a strong structure, which can for example be incorporated into a base using standard toothpaste mixing techniques, and which keeps the rigidity of the capsules on storage.

The alginate is typically present in an amount of up to about 5% by weight of the total weight of the encapsulate. Good results have been obtained at lower levels, eg constituting about 1% or less of the total weight of the encapsulate.

The weight ratio of active material to alginate is preferably in the range 5:1 to 300:1, more preferably 10:1 to 100:1, most preferably 20:1 to 60:1.

Where the active material is a solid, eg zinc salts such as zinc citrate trihydrate, the alginate and active material form a dispersion.

When the active material is a liquid, the alginate and active material form an emulsion, and are preferably formed into an emulsion by use of an emulsifying agent. The emulsifying material is preferably a water soluble polysaccharide, with a molecular weight of greater than 10,000. For example hydroxy cellulosic materials, a polysaccharide derived from the monosaccharide glucose, such as hydroxyethyl cellulose (HEC), hydroxypropyl cellulose (HPC) and hydroxypropylmethyl cellulose, or other polysaccharides containing different sugar monosaccharides where some of the primary alcohol groups of the polysaccharide have been oxidised to form uronic acid, for example D-glucuronic acid formed by the oxidation of the primary alcohol groups of glucose. Other examples include D-mannuronic acid and D-galacturonic acid. Such polysaccharides and their derivatives include Pectins, gum Tragacanth, alginates, propylene glycol alginate, gum Arabic and gum Ghatti. Combinations of different gums can also be used to achieve superior emulsion stability, such as the combination of propylene glycol alginate and pectins. These materials, particularly propylene glycol alginate, have good emulsifying properties and result in encapsulates with good stability.

The emulsifying agent preferably has a molecular weight (weight average) greater then 10,000, more preferably greater then 25,000, and particularly less than 2,000,000, and especially less than 1,000,000.

The emulsifying agent is typically present in an amount in the range from 0.2 to 5%, more preferably 0.5 to 2% and particularly 0.5 to 1% by weight of the total weight of the encapsulate.

The encapsulates preferably contain little or no materials which would act to solubilise the active material, such as most surfactants with a molecular weight of less than 5000, possibly resulting in leaching of the active material from the encapsulates over time. Some common surfactants such as Tween 20 (polyoxyethylene (20) sorbitan monolaurate ex ICI - Tween 20 is a Trade Mark) may have this effect, and so preferably should be avoided.

The balance of the encapsulate ingredients is usually water, preferably distilled water.

Alginate encapsulation is a known technique, and methods for producing encapsulates in accordance with the invention, eg using techniques discussed in the prior art mentioned above, are known to those skilled in the art.

In a further aspect, the invention provides a method of producing hydrated encapsulates of active material, comprising forming an emulsion or dispersion of alginate and active material, and causing cross-linking of the alginate to produce hydrated encapsulates.

The method conveniently involves mixing alginate, active material, emulsifying agent and water to produce an aqueous emulsion, eg using high shear mixing, and contacting droplets of the emulsion with an aqueous solution of those divalent or trivalent metal ions known to form insoluble gels, such as calcium and zinc, eg by spraying the emulsion into calcium chloride solution. Therefore, by preparing an emulsion of an alginate solution and dental flavour and adding this, drop wise, into a calcium chloride solution, spherical encapsulates are formed. Encapsulates of generally spherical form are produced virtually instantaneously by the known irreversible cross-linking reaction of alginate. If a chloride solution is used, the encapsulates are then preferably washed in water to remove chloride ions as these have a bitter taste, with the encapsulates then being suspended in water. Where other counterions such as lactate that do not have an unpleasant taste are used, washing is not required. The encapsulates should preferably be stored in hydrated form prior to use. Freeze drying or other drying techniques should preferably be avoided. The capsules can then be dosed into a toothpaste and rupture, releasing the flavour, on brushing.

By varying factors including emulsion viscosity, speed of spraying, droplet travel distance, droplet size etc in known manner, encapsulates of desired size can be produced. For most purposes, encapsulates having a diameter in the range 0.1 to 3mm, preferably 0.3 to 1mm, are suitable.

Encapsulate properties may also be affected to some extent by the time of contact with the calcium chloride or similar solution.

The encapsulates of the invention constitute a convenient delivery system for the active material and so find application in a number of uses including as ingredients in a range of water-based foodstuffs, confectionery, pharmaceuticals, cosmetic products and oral products.

In a further aspect the invention thus provides a water-based product, comprising encapsulates in accordance with the invention.

The encapsulates find particular application in toothpastes (or dentifrices) and other water-based oral products such as mouthwashes, and are particularly useful for delivery of dental flavours.

In a preferred aspect the invention thus provides a hydrated dental flavour encapsulate, comprising an emulsion or dispersion of dental flavour in an alginate matrix.

The invention may be used with a wide range of dental flavours, which are well known to those skilled in the art and are commonly mint-based. A mixture of dental flavours may be used.

The dental flavour enacapsulate conveniently also comprises an orally-compatible colouring material in the form of a pigment or oil soluble colour, eg titanium oxide for white colour, for aesthetic reasons to produce an attractive coloured product instead of a clear or colourless product.

The encapsulates may additionally or alternatively include active materials such as triclosan and zinc salts, eg zinc citrate trihydrate.

Encapsulates in accordance with the invention can be stable in water-based toothpastes on storage yet release the active material on brushing with no adverse mouthfeel effects, in a way that has not hitherto been possible. For maximum stability, the toothpaste preferably includes little or no anionic surfactant, eg sodium lauryl sulphate (SLS) which is commonly used in toothpaste, as anionic surfactants (with the possible exception of sodium cocoyl isethionate) tend to break down the encapsulates resulting in product instability and leaching of the active material. The toothpaste instead preferably includes one or more non-ionic surfactants and/or possibly one or more amphoteric surfactants, which do not break down the encapsulates in this way and so are not deleterious to

product stability. The criterion for stability seems to be approximately related to skin irritancy, ie the milder surfactants result in greater stability. Any non-ionic surfactant can be used for this purpose, including, for example, polyoxyethylene alkyl ethers, polyoxyethylene alklaryl ethers, polyoxyethylene-polyoxypropylene alkyl ethers, polyoxyethylene-polyoxypropylene block copolymers, sucrose fatty acid esters, glycerol fatty acid esters, propylene glycol fatty acid esters, sorbitan fatty acid esters, alkylglycoside fatty acid esters, polyoxyethylene sucrose fatty acid esters, polyoxyethylene glycol fatty acid esters, polyoxyethylene sorbitan fatty acid esters, alkylglycosides, fatty acid monoethanolamide, polyoxyethylene fatty acid monoethanolamide, polyoxyetheylene fatty acid diethanolamide, polyoxyethylene castor oils, poloxyethylene hydrogenated castor oils, and polyoxytheylene beeswax derivatives. A mixture of non-ionic surfactants may be used.

Amphoteric surfactants, such as cocoamidopropyl betaine, are also suitable for use in toothpastes.

The encapsulates in accordance with the invention are very rigid, post production, and can therefore be mixed into the toothpaste base using standard mixing techniques, and become weaker during storage in the product due to ion exchange reactions and loss of a proportion of the crosslinking ions to the non-ionic surfactant and any sequestrant in the product. However, the capsules remain intact during storage and give good mouthfeel effects on brushing, requiring little shear to release the active material.

The capsules can become unstable in the presence of anionic surfactants due to the removal of most of the crosslinking ions from the capsule system as the presence of any anion which will remove divalent ions by precipitation or sequestration will tend to solubilise the alginate. In the case of the capsules in accordance with this invention, this results in flavour/fragrance leaching and/or capsule dissolution since the corresponding calcium salts of anionic surfactants tend to be insoluble in water, therefore, precipitating out and become ineffective and cause clouding of the product.

However, sodium lauryl sulphate (SLS) is commonly used in toothpaste due to its high foaming efficiency, which can be difficult to achieve using non-ionic surfactants, and therefore, if the encapsulates are to be used in water based toothpaste containing SLS, a stabilising system is needed to reduce the amount of free monomer surfactant in the product. The preferred stabilising system for the encapsulates is an electrolyte/counterion

which decreases the critical micelle concentration of the surfactant by partially neutralising the anionic charge, decreasing the charge density. The electrolyte is preferably a sodium, potassium, zinc, or magnesium salt, more preferably a magnesium salt, particularly magnesium sulphate, which can be added to the product formulation. The concentration of the electrolyte is preferably in the range from 0.2 to 5%, more preferably 0.5 to 3%, and particularly 1 to 2% by weight of the total weight of the toothpaste.

The encapsulate stability in anionic surfactants is dramatically increased by the inclusion of, for example, 2% magnesium sulphate which not only improves the stability of the capsules by reducing the leaching of active materials but also improves the clarity of the product as less of the surfactant is precipitated out as calcium lauryl sulphate. The encapsulates may also decrease in size as they undergo their natural syneresis process, which can be regarded as a continuation of the process of gelation, and in consequence, they become more rigid. Thus, the encapsulates require more shear to rupture and release the entrapped active, however, this does not impart any adverse mouthfeel effects.

Most electrolytes appear to increase the stability of the encapsulates to some extent, for example tests carried out with sodium monofluorophosphate and sodium hydrogen carbonate showed that both the electrolytes increased the stability of the capsules in a 2% SLS solution when compared to a capsule sample containing no electrolyte. However, none of the electrolytes tested achieved the level of stability gained from incorporating magnesium sulphate. The use of magnesium sulphate also has the added advantage of being orally acceptable.

Another method of increasing capsule stability is to add a further additive to the capsule formulation to increase the barrier properties of the system. These include gums which are crosslinked by other ions such as Carrageenan (cross-linked with calcium and potassium ions) or Gellan gum (cross-linked with potassium, sodium, calcium and magnesium ions) or the addition of colloidal silica to the formulation, which not only increases the barrier properties but also acts as an emulsifier. Alternatively the addition of a gum which interacts with the alginate itself can be added to the formulation such as sodium carboxymethyl cellulose. These methods are effective at preventing the dissolution of the capsule in some anionic formulations, however, they only delay the leaching process.

The toothpaste may be otherwise of generally conventional formulation, eg as disclosed in EP 0747037. Additional possibilities not mentioned in EP 0747037 include the following:

Additional humectant - xylitol
Additional thickener - xanthan gum
Any suitable flavour - can include both natural and/or synthetic
Additional sweetening agents - aspartame, acesultame K, talin
Additional anti-plaque agent -cetyl pyridinium chloride
Use of peroxides and other whitening agents
pH range 5.5 to 9.0

The encapsulates can be readily incorporated into standard toothpaste bases using conventional toothpaste mixing techniques.

In a preferred aspect, the invention thus provides a water-based toothpaste comprising encapsulates in accordance with the invention and preferably containing substantially no anionic surfactant. The encapsulates conveniently include a dental flavour.

In another preferred aspect, the invention provides a water-based toothpaste comprising encapsulates in accordance with the invention and contains an anionic surfactant and stabilising system such as magnesium sulphate.

The use of high weight ratios of dental flavour to alginate, as noted above, gives a cost-effective toothpaste with good flavour release and mouthfeel properties in use.

As noted above, encapsulates in accordance with the invention can be stable in toothpastes so that the active material, eg dental flavour, remains encapsulated in the toothpaste on storage. The encapsulates will normally be colourless and so not be visually apparent in the toothpaste, however if the encapsulates include a colouring material or dye they will be visible in the toothpaste possibly providing a pleasing visual effect that will be readily apparent if the toothpaste is presented in a transparent package.

The alginate encapsulates weaken and rupture easily, eg on dispensing or use, while nevertheless remaining intact on storage. If rupturing of coloured encapsulates occurs on dispensing, eg squeezing the toothpaste from a tube, this may result in formation of stripes in the toothpaste, which again may be visually pleasing. The encapsulates will in any

event rupture on use, eg on brushing, with consequential release of contents. This opens the possibility of creating novel flavour effects, such as change of flavour on brushing.

Comparative tests have shown that a given amount of flavouring material in a toothpaste produces a stronger effect when in the form of encapsulates in accordance with the invention, thus providing possibilities for reducing flavouring material usage in toothpaste, with consequential cost savings, without loss of flavour effects.

The invention will be further described, by way of illustration, in the following Examples and with reference to the accompanying drawing in which:

Figure 1 is a taste chart comparing the effects of toothpaste flavouring in the form of encapsulates in accordance with the invention with toothpaste containing the same flavouring at the same level but not in encapsulated form.

Example 1

Dental flavour encapsulates in accordance with the invention were prepared by mixing using a high shear Silverson mixer the following ingredients (amounts specified in % by weight) to form an emulsion.

Sodium Alginate (Manugel DMB ex Nutrasweet Kelco)	0.8%
HEC (*Natrosol 250LR ex Aqualon)	0.5%
Distilled Water	74.7%
Dental Flavour A (Peppermint)	25.0%

* Natrosol is a Trade Mark.

The composition of dental flavour A is as follows:

	Weight %
1-methoxy-4-propenylbenzene	8.6
Clove Bud Oil	0.3
Eucalyptol	2.7
Lemon Oil	0.5
3-hydroxy-2-methyl-4-pyrone	0.1

Menthol laevo	28.7
Orange Oil	0.2
Pepper Black Oil	0.1
Peppermint Oil	58.8
Total	100.0

The emulsion was sprayed as droplets into a reservoir of aqueous calcium chloride, resulting in irreversible cross-linking of the alginate to produce generally spherical encapsulates. The encapsulates were removed from the calcium chloride solution after an appropriate residence time (approximately sixty seconds) to produce encapsulates having desired features. The encapsulates were washed in distilled water to remove free chloride ions that would cause a bitter taste in the final product, and suspended in distilled water for storage. The encapsulates had a diameter of about 1mm in the present case, obtained in known manner by regulating factors including emulsion viscosity, spray pump speed, spray nozzle size and distance from spray nozzle to calcium chloride solution.

The encapsulates can be stored in hydrated form for extended periods of time (at least 6 months) without significant loss of flavour. In contrast, freeze-drying of the encapsulates resulted in substantial and immediate loss of flavour.

The encapsulates were incorporated into a toothpaste formulation/mixer T1 as specified below, in an amount for the dental flavour to constitute 1% by weight of the total weight of the toothpaste, using standard toothpaste mixing techniques. The toothpaste base was that of a generally conventional water-based toothpaste but including the non-ionic detergent Lutrol F127 (Lutrol F127 is a polyoxyethylene-polyoxypropylene block copolymer ex BASF-Lutrol is a Trade Mark) in place of more commonly used anionic surfactants. The toothpaste includes no anionic surfactants. The encapsulates are incorporated intact in the toothpaste and remain stable in the toothpaste for extended periods (at least 6 months).

Toothpaste T1

	Weight %
Sorbitol (70% solution)	61.6
Water (mains)	13.15

Sodium monofluorophosphate	0.8
Trisodium phosphate anhydrous	0.1
Silica thickener	8.0
Silica abrasive	8.0
Sodium carboxymethyl cellulose	0.55
Lutrol F127	2.0
Polyethyleneglycol 1500	4.0
Saccharin (25%aq)	0.8

The capsules were mixed on a standard Fryma toothpaste mixer.

Sensory evaluation tests were carried out by a trained sensory panel using toothpaste prepared generally as described above containing 1% by weight of flavour in encapsulates in accordance with the invention, and similar toothpaste containing 1% of the same flavour material in free form, ie not encapsulated. The results are shown in the taste chart of Figure 1, and demonstrate significant improvements in flavour impact by using encapsulates in accordance with the invention. Improvements in longevity of the flavour were also detected.

Example 2

Dental flavour encapsulates were prepared by making an emulsion of the following ingredients using a Silverson mixer.

	Weight %
Sodium Alginate (Manugel DMB)	0.8
HEC (Natrosol 250LR)	0.2
Distilled water	73.0
Dental Flavour A	25.0
Titanium Dioxide	1.0

The emulsion was sprayed into a reservoir of aqueous calcium chloride and washed with distilled water as detailed in Example 1. The encapsulates were then incorporated into the toothpaste formulation (T1) in an amount to constitute 1% flavour by weight, using standard toothpaste mixing techniques (a Fryma mixer). A control sample was also

prepared at the same time containing 1% of the same flavour, not encapsulated. The samples were then stored at 0°C and 45°C in standard toothpaste tubes for six weeks.

After this period the total flavour content of the toothpaste sample was quantified using standard solvent extraction techniques and analysed using Gas Chromatography. The total flavour content was based on the percentage of menthone, iso-menthone and menthol remaining in the samples compared to standards.

The following Table indicates the total flavour content of the two samples based on three ingredients.

% Ingredient	Encapsulated		Free Flavour	
	0°C	45°C	0°C	45°C
Menthone	1.07	1.01	0.93	0.69
Iso-menthone	0.99	0.99	0.91	0.70
Menthol	1.02	1.07	1.03	0.84
Total	1.03	1.05	1.00	0.80

Using the above formulation the encapsulates remained intact on squeezing the toothpaste from the tube and only ruptured, releasing the flavour, on brushing.

Example 3

Dental Flavour encapsulates were prepared by making an emulsion of the following ingredients using a Silverson mixer:

	Weight %
Sodium Alginate (Manucol DM ex Nutrasweet Kelco)	1.0
HEC (Natrosol 250LR)	0.5
Distilled water	68.5
Dental Flavour A*	30.0

* Containing 0.1% D&C Red # 17

The alginate Manucol DM (Manucol is a Trade Mark) contains a greater proportion of polymannuronic acid to polyguluronic acid units as compared with Manugel DMB. The emulsion was sprayed into a reservoir of aqueous calcium chloride and washed with distilled water as detailed in Example 1. The encapsulates were then incorporated into the toothpaste formulation T1 in an amount to provide 1% flavour by weight, using standard toothpaste mixing techniques (Fryma mixer). The toothpaste was then stored in standard toothpaste tubes, and on squeezing out the toothpaste the encapsulates smeared into stripes of a pleasing aesthetic appearance.

Example 4

Dental flavour encapsulates were prepared by making an emulsion of the following ingredients using a Silverson:

	Weight %
Sodium Alginate (Manugel DMB)	0.8
PGA* (Kelcoloid S ex NutraSweet Kelco)	0.4
Distilled water	72.8
Dental Flavour A	25.0
Titanium Dioxide	1.0

*Propylene Glycol Alginate
Sodium Alginate ex NutraSweet Kelco

The emulsion was sprayed into a reservoir of aqueous calcium chloride and washed with distilled water as detailed in example 1. The encapsulates were then incorporated into the toothpaste formulation (T2) in an amount to constitute 1% flavour by weight, using standard toothpaste mixing techniques.

The capsules dosed into the toothpaste formulation (T2) were found to remained intact after storage at 45°C for four months with only slight leaching of the flavour. Stability is improved in the presence of the anionic detergent due to the addition of magnesium sulphate to the product formulation which decrease the critical micelle concentration of the surfactant, resulting in less free surfactant to destabilise the capsule system.

Toothpaste T2

	Weight %
Sorbitol (70% solution)	60.6
Water (mains)	12.15
Sodium monofluorophosphate	0.8
Trisodium phosphate anhydrous	0.1
Magnesium sulphate anhydrous	2.0
Silica thickener	8.0
Silica abrasive	8.0
Sodium carboxymethyl cellulose	0.55
Empicol LZPV/C	2.0
Polyethyleneglycol 1500	4.0
Saccharin (25% aq)	0.8

Example 5

Dental flavour encapsulates were prepared by making an emulsion of the following ingredients using a Silverson:

	Weight %
Sodium Alginate (Manugel DMB)	0.8
PGA* (Kelcoloid S ex NutraSweet Kelco)	0.4
Pectin (No. P-2157 ex Sigma)	0.4
Distilled water	57.4
Dental Flavour A	40.0
Titanium Dioxide	1.0

*Propylene Glycol Alginate
Sodium Alginate ex NutraSweet Kelco

The emulsion was sprayed into a reservoir of aqueous calcium chloride and washed with distilled water as detailed in example 1. The combination of gums was found to give stable capsules with flavour loadings as high as 40%.

Example 6

Dental flavour encapsulates were prepared by making an emulsion of the following ingredients using a Silverson:

	Weight %
Sodium Alginate (Manugel DMB)	0.4
Gellan Gum (Kelcogel F)	0.4
Distilled water	64.0
Colloidal silica (Ludox HS 40)	10.0
PGA (Kelcoloid S)	0.2
Dental Flavour A*	25.0

* Containing 0.1% Phthalocyanine Blue 22005 ex Anstead International
Sodium Alginate ex NutraSweet Kelco
Polyvinyl alcohol supplied by British Traders
Gellan Gum (Kelcogel F) ex NutraSweet Kelco
Colloidal silica (Ludox) ex Du Pont
PGA: Propylene Glycol Alginate(Kelcoloid S ex NutraSweet Kelco)

The emulsion was sprayed into a reservoir of aqueous calcium chloride and washed with distilled water as detailed in example 1. The encapsulates were then incorporated into the toothpaste formulation (T3) in an amount to constitute 1% flavour by weight, using standard toothpaste mixing techniques. The combination of gums was found to give increased capsule stability in toothpaste containing anionic detergents without the use of magnesium sulphate.

Toothpaste T3

	Weight %
Sorbitol (70% solution)	61.6
Water (mains)	13.15
Sodium monofluorophosphate	0.8
Trisodium phosphate anhydrous	0.1
Silica thickener	8.0
Silica abrasive	8.0
Sodium carboxymethyl cellulose	0.55

Empicol LZPV/C	2.0
Polyethyleneglycol 1500	4.0
Saccharin (25% aq)	0.8

Empicol LZPV/C - ex Albright & Wilson Detergents Divison

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CLAIMS

1. A hydrated encapsulate of active material, comprising an emulsion or dispersion of active material in an alginate matrix.

2. An encapsulate according to claim 1, wherein the active material is selected from flavourings, fragrances, colourings and antibacterial agents.

3. An encapsulate according to claim 1 or 2, wherein the active material is present in an amount up to 60%, preferably 10 to 60%, more preferably 20 to 60%, most preferably 25 to 30%, by weight of the total weight of the encapsulate.

4. An encapsulate according to any one of the preceding claims, wherein the alginate has a high percentage of polyguluronic acid units.

5. An encapsulate according to any one of the preceding claims, wherein the alginate is present in an amount up to 5%, typically about 1%, by weight of the total weight of the encapsulate.

6. An encapsulate according to any one of the preceding claims, wherein the weight ratio of active material to alginate is in the range 5:1 to 300:1, preferably 10:1 to 100:1, more preferably 20:1 to 60:1.

7. An encapsulate according to any one of the preceding claims, further comprising a water soluble polysaccharide, preferably having a molecular weight of greater than 10,000, as an emulsifying agent.

8. An encapsulate according to claim 7, wherein the emulsifying agent is selected from (i) hydroxy cellulosic materials such as hydroxyethyl cellulose, hydroxypropyl cellulose and hydroxypropylmethyl cellulose, and (ii) other polysaccharides such as Pectins, gum Tragacanth. propylene glycol alginate, gum Arabic and gum Ghatti.

9. An encapsulate according to claim 7 or 8, wherein the emulsifying agent is present in an amount up to about 1%, typically about 0.5%, by weight of the total weight of the encapsulate.

10. An encapsulate according to any one of the preceding claims, further comprising a water soluble polysaccharide, which can be crosslinked with different monovalent, ddivalent and trivalent ions, such as carrageenan, gellan gum and carboxymethyl cellulose.

11. An encapsulate according to any one of the preceding claims, further comprising a colloidal silica.

12. An encapsulate according to any one of the preceding claims, having a diameter in the range 0.1 to 3mm, preferably 0.3 to 1mm.

13. A method of producing hydrated encapsulates of active material, comprising forming an emulsion or dispersion of alginate and active material, and causing cross-linking of the alginate to produce hydrated encapsulates.

14. A method according to claim 13, wherein droplets of the emulsion or dispersion are contacted with an aqueous solution of divalent or trivalent metal ions.

15. A water-based product comprising encapsulates in accordance with any one of claims 1 to 12.

16. A product according to claim 15, comprising toothpaste.

17. A product according to claim 16, wherein the active material comprises dental flavour.

18. A product according to claim 16 or 17, wherein the active material comprises colouring agent.

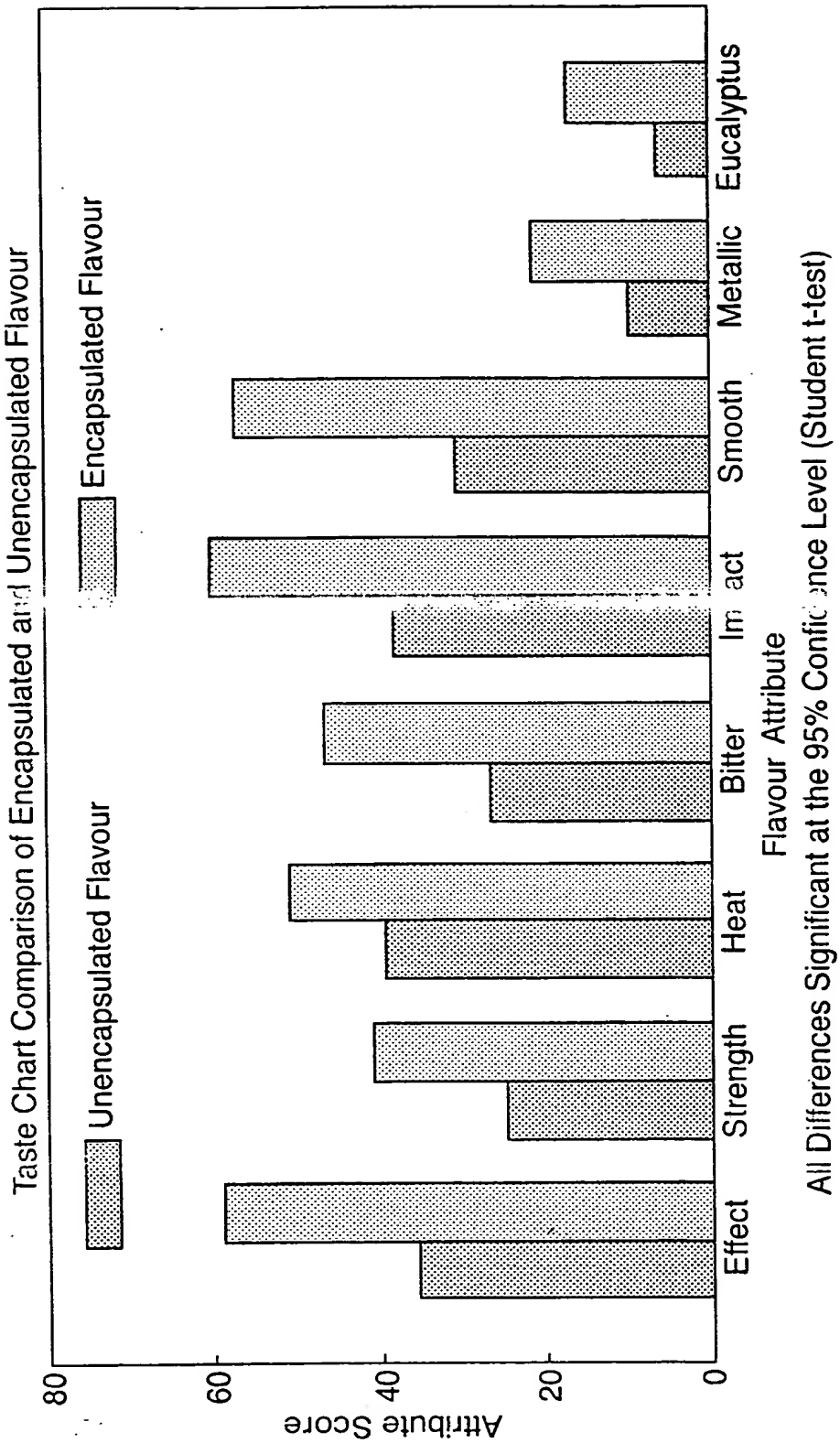
19. A product according to claims 16, 17 and 18, comprising non-ionic surfactant and substantially no anionic surfactant.

20. A product according to claims 16, 17 and 18, comprising an anionic surfactant and an electrolyte.

21. A product according to claim 20, wherein the electrolyte is selected from sodium, potassium, zinc, or magnesium salt.

22. A product according to claim 21, wherein the electrolyte is magnesium sulphate.

Fig.1.



INTERNATIONAL SEARCH REPORT

Application No
PCT/GB 98/03345

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 B01J13/08 A61K7/16 A61K7/00 A23L1/22 A23L1/27

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A61K B01J A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 929 403 A (THE UPJOHN CO.) 19 June 1963 see page 2, line 13 - line 88 see page 3, line 9	1,2
A	WO 96 40069 A (STANFORD RES INST INT) 19 December 1996 see page 5, line 10 - line 28	1,3
X	see claims 25-28	11,12
X	DATABASE WPI Section Ch, Week 8010 Derwent Publications Ltd., London, GB; Class A11, AN 80-17311C XP002059394 & JP 55 011029 A (KIBUN KK) , 25 January 1980 see abstract	1,2

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
4 February 1999	11/02/1999

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

Int[redacted] Application No
PCT/GB 98/03345

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	EP 0 391 803 A (L'OREAL) 10 October 1990 see claims 1,2	1,4,11, 12
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